

टार और बिटुमिनस सामग्री — परीक्षण
पद्धतियाँ — ट्राइक्लोरोइथिलीन में
घुलनशीलता का निर्धारण

(दूसरा पुनरीक्षण)

Tar and Bituminous Materials —
Methods for Test — Determination of
Solubility in Trichloroethylene

(Second Revision)

ICS 75.140

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Bitumen, Tar and Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1958 as 'Methods for testing tar and bituminous materials — Determination of solubility in carbon disulphide or trichloroethylene' and subsequently revised in 1978. A series of published Indian Standards cover tar, bitumen and bituminous materials and a booklet was published containing a series of 22 standards as listed below:

<i>IS No.</i>	<i>Title</i>
IS 1201 : 1978	Sampling
IS 1202 : 1978	Determination of specific gravity
IS 1203 : 1978	Determination of penetration
IS 1204 : 1978	Determination of residue of specified penetration
IS 1205 : 1978	Determination of softening point
IS 1206 : 1978	Determination of viscosity:
(Part 1)	Industrial viscosity
(Part 2)	Absolute viscosity
(Part 3)	Part 3 Kinematic viscosity
IS 1207 : 1978	Determination of equiviscous temperature (EVT)
IS 1208 : 1978	Determination of ductility
IS 1209 : 1978	Determination of flash point and fire point
IS 1210 : 1978	Float test
IS 1211 : 1978	Determination of water content (dean and stark method)
IS 1212 : 1978	Determination of loss on heating
IS 1213 : 1978	Distillation test
IS 1214 : 1978	Determination of matter insoluble in benzene (WITHDRAWN due to toxic nature of benzene)
IS 1215 : 1978	Determination of matter insoluble in toluene
IS 1216 : 1978	Determination of solubility in carbon disulphide or trichloroethylene
IS 1217 : 1978	Determination of mineral matter (ash)
IS 1218 : 1978	Determination of phenols
IS 1219 : 1978	Determination of naphthalene
IS 1220 : 1978	Determination of volatile matter content

However, the Committee responsible for the formulation of standards in the field of bitumen, tar and related products decided to publish these Indian Standards separately for each test so as to make it user friendly.

(Continued on third cover)

Indian Standard

TAR AND BITUMINOUS MATERIALS — METHODS FOR TEST — DETERMINATION OF SOLUBILITY IN TRICHLOROETHYLENE

*(Second Revision)***1 SCOPE**

This standard prescribes the method of test for determination of solubility in trichloroethylene for asphaltic bitumen and native asphalts.

2 REFERENCES

The standards given below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All the standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

<i>IS No.</i>	<i>Title</i>
IS 245 : 2020	Specification for trichloroethylene, technical (<i>fourth revision</i>)
IS 334 : 2023	Glossary of terms relating to bitumen and tar (<i>fourth revision</i>)
IS 1211 : 2022	Methods for testing tar and bituminous materials — Determination of water content — Dean and stark method (<i>second revision</i>)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 334 shall apply.

4 METHOD A (FOR ASPHALTIC BITUMEN)**4.1 Apparatus**

4.1.1 Balance — capable to weigh with the least count of 0.001 g.

4.1.2 Oven

4.1.3 Gooch Crucible

4.1.4 Conical Glass Flask — 200 ml capacity

4.2 Solvent

4.2.1 Trichloroethylene — (*see* IS 245)

4.3 Preparation of Material

If the sample contains water, heat it to a temperature not exceeding 130 °C until the water has been removed, stirring constantly, when possible, during heating. Perform this operation as expeditiously as possible.

4.4 Preparation of Gooch Crucible

4.4.1 Insert the filter tube in the stopper of the filtering flask, set the Gooch crucible in the filter tube, and connect the flask to the suction pump. Fill the crucible with some of the suspension of asbestos or glass microfiber in the water, allow it to settle partly in the crucible and apply a light suction to draw off the water, leaving a firm mat of asbestos or glass microfiber in the crucible. Add more suspended asbestos or glass microfiber and repeat the process until a mat weighing (0.5 ± 0.1) g is built up after drying. Wash the asbestos or glass microfiber mat thoroughly with water and dry in the oven at a temperature of 150 °C. Cool the crucible in the desiccator, weigh and replace it in the dry filter tube supported in the clean and dry filtering flask.

4.4.2 In the determination, the asbestos or glass microfiber apparently absorbs irreversibly an amount of soluble bitumen (usually 1 mg/g to 5 mg/g of asbestos or glass microfiber) which is not removed by a subsequent washing with solvent. The weight of asbestos used, therefore, shall be kept within the specified limits to ensure reproducible results.

4.5 Procedure

Weigh about 2 g of the dry sample correct to the nearest 0.001 g into a 200 ml conical flask and add 100 ml of trichloroethylene. Stir the contents of the flask, and then allow it to stand, loosely corked, for a period of one hour. Filter the contents of the flask through the Gooch crucible prepared as described under 4.4 which has been weighed to the nearest 0.001 g. Moisten the filter pad with trichloroethylene before commencing filtration and filter at a rate of not

more than two drops per second at first. The filtrate shall be quite clear. Transfer the insoluble matter remaining in the flask to the crucible by washing out the flask with a stream of trichloroethylene from a wash bottle. Wash the sample retained in the crucible with successive small amounts of trichloroethylene until a filtrate is obtained which is not discoloured. Allow the crucible to dry in air for 30 min, after which place it in an oven at 100 °C to 110 °C for one hour. Allow the crucible to cool in a desiccator and then weigh.

4.6 Calculation

Calculate the matter soluble in trichloroethylene as follows:

Matter soluble in trichloroethylene, percent

$$= \frac{W_1 - W_2}{W_1} \times 100$$

where

W_1 = mass, in g, of dry sample taken for the test;
and

W_2 = mass, in g, of insoluble material retained in the Gooch crucible.

4.7 Report

Report the result obtained in 4.6 to nearest 0.05 percent as the matter soluble in trichloroethylene of the dry material.

4.8 Precision

Test results shall not differ from the mean by more

than the following:

<i>Sl No.</i>	<i>Matter Soluble in Trichloroethylene</i>	<i>Repeatability</i>	<i>Reproducibility</i>
(1)	(2)	(3)	(4)
i)	Below 98 percent	0.5	1.0
ii)	98 to 100 percent	0.1	0.2

5 METHOD B (FOR NATIVE ASPHALTS)

5.1 Apparatus

5.1.1 Glass Tap Funnel — approximately 100 mm diameter, the stem fitted with a tap and the top ground flat.

5.1.2 Glass Plate — to cover the funnel, about 120 mm in diameter, with a hole of about 16 mm diameter in the centre.

5.1.3 Glass Funnel — smaller than that described under 5.1.1 with its stem passing through a cork placed in the hole in the glass plate, the lower end of the stem reaching approximately half-way down the tap funnel.

5.1.4 Filter Papers — two, Whatman filter paper No. 5 or equivalent filter papers, 185 mm in diameter, dried in a suitable oven, cooled in a desiccator, counterpoised and folded together as shown in Fig. 1.

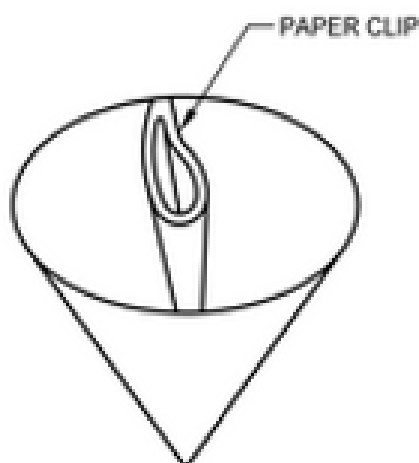


FIG. FOLDED FILTER PAPER

5.2 Solvent and Sample

5.2.1 Trichloroethylene (*see* IS 245)

5.2.2 Cement

Suitable for sealing the glass plate to the funnel. It may be prepared with 10 g of gelatin, 80 ml of water and 20 g of glycerine.

5.3 Preparation of Sample

5.3.1 If the sample contains water, heat a 100 g portion at a temperature not exceeding 130 °C, the material being constantly stirred when possible, until the rate of loss in weight of the sample does not exceed 0.1 g during a 15 min period of heating.

5.3.2 If the loss in weight on drying exceeds 0.1 percent, determine the water content of another sample of the material in accordance with IS 1211.

5.4 Procedure

Dry the two filter papers (*see* 5.1.4) in an oven at 100 °C to 110 °C, cool in a desiccator, counterpoise, fold together as illustrated in Fig. 1 and place in the tap funnel. Weigh about 2 g of the dry sample, as prepared in 5.3, correct to the nearest 0.001 g in the filter papers and secure the cover to the funnel by means of the cement. Now add trichloroethylene through the small funnel until the filter paper is about half filled and then allow it to stand for 30 min. Draw the solution off through the tap. Place a watch glass on the small funnel to minimize evaporation. Close the tap and introduce a further quantity of trichloroethylene through the small funnel and again draw the solution off after 30 min. Repeat this procedure until the solvent drawn off is no longer discoloured. When the filtration is completed, remove the cover and allow the filter papers to dry in air for 30 min before placing them in a ventilated air oven at 100 °C to 110 °C for 1 h. Next place the filter papers in a desiccator and when cool, weigh the inner filter paper and contents, using the outer filter paper as a counterpoise.

5.5 Calculation

Calculate the solubility of the dry sample as follows:

Matter soluble in trichloroethylene =

$$\left(\frac{W_1 - W_2}{W_1} \times 100 \right) \times \frac{100}{100 + W_3}$$

percent by weight

where

W_1 = mass, in g, of the dry sample taken for the test;

W_2 = mass, in g, of recovered insoluble matter; and

W_3 = water content of sample, in g, if determined.

5.6 Report

Report the result obtained in 5.5 to the nearest 0.05 percent as the matter soluble in trichloroethylene.

5.7 Precision

Test results shall not differ from the mean by more than the following:

Repeatability	0.5
Reproducibility	0.5

These precision figures do not include any allowance for differences due to variations in samples.

6 PRECAUTIONS

6.1 Barriers

Handling of trichloroethylene shall be done in fume cupboard with ventilation.

6.2 Personal Protective Equipment (PPE)

Chemical gloves, eye goggles, face shield, spill proof clothing, chemically resistant safety shoes shall be provided.

6.3 Safety shower nearby and eye wash in immediate vicinity shall be provided.

NOTE — It is strongly recommended that the safety in-charge reviews the safety data sheet (SDS) of these materials and setup the operating procedure, PPE and barriers accordingly.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Bitumen, Tar and Related Products Sectional Committee, PCD 06

<i>Organization</i>	<i>Representative(s)</i>
CSIR - Central Road Research Institute, New Delhi	PROF MANORANJAN PARIDA (Chairperson)
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G R Infra-Projects Limited, Gurugram	SHRI U. C. GUPTA

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Indian Institute of Technology Madras, Chennai	DR ATUL NARAYANAN
Indian Institute of Technology Roorkee, Roorkee	SHRI SHAM SUNDAR RAVINDRANATH
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Indian Oil Corporation Limited - Refineries and Pipelines Division, New Delhi	SHRI ANINDITA MOITRA SHRI SOUMEN MONDAL (<i>Alternate I</i>) SHRI P. MADHUSUDHANA REDDY (<i>Alternate II</i>)
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BIS Directorate General	SHRIMATI MEENAL PASSI, SCIENTIST 'F'/ SENIOR DIRECTOR AND HEAD (PETROLEUM, COAL, AND RELATED PRODUCTS)[REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary
SHRI HARI MOHAN MEENA
SCIENTIST C/DEPUTY DIRECTOR
(PETROLEUM , COAL, AND RELATED PRODUCTS), BIS

(Continued from second cover)

This second revision has been brought to formulate individual standard on determination of solubility in trichloroethylene. In this revision following major changes have been made:

- a) Use of carbon disulphide has been removed from the test method due to its carcinogenicity; and
- b) Precautions for use of trichloroethylene have been incorporated.

The composition of the Committee responsible for formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

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This Indian Standard has been developed from Doc No.:PCD 06 (19995).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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